Extraction and Characterization of Silica from Reeds Biomass (Imperata cylindrica) in Various Annealing Temperatures

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Abstract—Reeds biomass has been successfully extracted. It calcined with annealing temperatures at 800°C (A800), 850°C (A850), and 900°C (A900). The x-ray pattern showed that it has a cristoballite (SiO2) crystal structure. The band gap energy values are 3.8 eV, 3.7 eV and 4.7 eV, respectively. FTIR spectra show the groups are silanol, siloxane, and mono hydrate. Quantitatively, it provides results to determine the optical properties and dielectric functions which indicate a shift to longer wavelengths with an increase in annealing temperature. The morphology presents a different image where the particles are formed flakes (A800), aggregates (A850) and porous aggregates (A900).

Keywords: Reeds biomass, annealing temperature, cristoballite, band gap energy, optical properties, dielectric function

In Indonesia, reeds (Imperata cylindrica) are natural vegetation that is very abundant and covers a total of 8.5 million hectares, especially in tropical and subtropical areas. Reeds plants are wild plants and easy to grow and reproduce [1]. Reeds are also known as some of the most difficult to eradicate and harmful pest plants. People use reeds for making briquettes and roofs, but their use is limited [2]. The roots are used as medicine, and the leaves are disposed of as agro-waste. The ash from the leaves of reeds can be used as a material for making silica. Silicon dioxide/Silica (SiO2) is a crucial ingredient in manufacturing a wide variety of materials [3], such as cement and ceramics [4], textiles [5], films [6], paper [7], rubber [8], tires [9]. In this study, we annealed reeds biomass with various annealing temperatures. Annealing is a heat treatment process that changes the physical and sometimes also the chemical properties of a material to increase ductility and reduce the hardness to make it more workable [3], [10], [11].

Figure 1 shows the XRD patterns of annealed reeds biomass at A800, A850, and A900. Qualitative analysis using Match! software confirms that the XRD patterns are SiO2 (cristoballite) (ICDD#96-900-8230). An increase in annealing temperature causes a slight shift in the peak position, addition of peaks, and sharper peak, marked by the values of 20 (see the data in Table 1).

The band gap energy is calculated using the Direct Tauc plot method. It determines the optical band gap by looking at a linear graph of the relationship E (eV) on the x-axis and (ahv)1/2 y-axis. In Fig. 2, the temperature increase from A800 to A850 shows the decrease of band gap energy values from 3.8 eV to 3.7 eV, and at A900, it increases significantly to 4.7 eV. This shift occurs because a higher annealing temperature can produce higher electron excitation from the valence band to the conduction band as a direct transition.

Figure 3 shows the FTIR spectra of annealed reeds biomass at A800, A850, and A900, the functional groups are indicated by numbers and summarized in Table 2. The functional groups that appear are Si-O-Si, Si-Si, Si-O, Si-OH, H-Si-O-H, and Si-OH.

The FTIR pattern was used to determine the optical properties and dielectric function for the firm peaks at the wavenumbers (ω) from 400 to 550 cm−1 [12]. For these purposes, Kramers–Kronig’s (KK) relation is used in the quantitative analysis of the FTIR pattern. The FTIR spectra were used for determining the refractive index (n), extinction coefficient (k) are shown in Fig. 4(a). The lower intersection wavelength point between n(ω) and k(ω) is the transverse optical (To) mode, and the higher one is the longitudinal optical (Lo) phonon vibration mode. The

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values of $T_o$ and $L_o$ serve in Table 3. The values of $T_o$ generally increase with the increasing of annealing temperature, and the values of $L_o$ at A900 significantly increase because the increasing of annealing temperature can produce higher vibration.

The dielectric functions for the real part $\varepsilon_1(\omega)$ and imaginary part $\varepsilon_2(\omega)$ in Fig. 4(b) indicate by peaks in the range from 430 to 450 cm$^{-1}$, it means that at the surface layer, the breaking of the interatomic bonding happens, and a new structure is formed. $\Delta(L_o-T_o)$ is the distance between two optical phonon modes, which increases on increasing annealing temperature as presented in Table 3, probably due to a less stable structure and nonuniformity of lattice in the annealed reeds biomass [13–14].

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Figure 5 shows the morphology of annealed reeds biomass at A800, A850, and A900 with magnification (a) 2500× and (a) 5000×. The particle with magnification 2500× (Fig. 5(a) A800) shows an irregular flakes form, aggregates form (Fig. 5(a) A850) and porous aggregates form (Fig. 5(a) A900). Then, magnification 5000× in Fig. 5(b) A800, A850, and A900 clearly represent that the increasing of annealing temperature causes a pores form on the surface of reeds biomass particles.

Table 3. Transverse optical phonon (To) and longitudinal optical phonon (Lo), and the real (ε₁) and imaginary parts (ε₂) of the dielectric function from the quantitative analysis of the FTIR spectra in Figure 4 (a) and (b).

<table>
<thead>
<tr>
<th>Samples</th>
<th>Tₒ (cm⁻¹)</th>
<th>Lₒ (cm⁻¹)</th>
<th>Δ(Lₒ–Tₒ) (cm⁻¹)</th>
<th>ε₁</th>
<th>ε₂ (cm⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A800</td>
<td>403.122</td>
<td>538.139</td>
<td>135.017</td>
<td>403.122</td>
<td></td>
</tr>
<tr>
<td>A850</td>
<td>403.122</td>
<td>540.068</td>
<td>136.946</td>
<td>405.051</td>
<td></td>
</tr>
<tr>
<td>A900</td>
<td>416.624</td>
<td>601.790</td>
<td>185.166</td>
<td>416.624</td>
<td></td>
</tr>
</tbody>
</table>

Fig. 5. The morphology result of annealed reeds biomass at A800, A850, and A900 with magnification (a) 2500× and (b) 5000×

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